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1,002,786

# PATENT SPECIFICATION

NO DRAWINGS

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## COMPLETE SPECIFICATION

### Process of producing Foamed Glass and Foamed Glass and Insulating Material produced by this process

I, WERNER HERMANN KREIDL, personally responsible partner of KREIDL KOMMANDIT-GESELLSCHAFT, a citizen of the United States of America, residing at 601, Raditsch, Vaduz, Liechtenstein, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The main patent Application No. 16582/62 (Serial No. 1,000,136) relates to insulating material formed as glassy solidified mass with a foam structure in which the walls of communicating or closed macrocells have a closed microcellular structure.

The term "microcells" as used herein means cells which may be up to 1/10 (e.g. from 1/100 to 1/1000) of the diameter of the macrocells.

These macrocells are like the cells which are present in conventional foamed glass. They are separated from each other by relatively thin cell walls and constitute the foamed structure. The fact that microcells are present in the internal cell walls separating these macrocells results in essential advantages. Glass or glasslike material is hard and brittle. For this reason the usual foamed glass, in which the cells are separated from each other by solid cell walls, can be machined only with difficulty, and then only if the cell walls between the pores are relatively thin. In these cases, the compressive strength is low. For this reason the usual foamed glass must generally be made in moulds which correspond to the desired shape of the foamed glass body. For this reason, a large number of complicated moulds must be manufactured and kept in stock. In the material having micro-

cells present in the macrocell walls, these microcells may be considered points of intentional weakness in the macrocell walls. It has been found that these microcells in the macrocell walls impart to the material a very good machinability in sawing, drilling and boring, milling and the like operations. Hence, this material enables the desired bodies of foamed glass or the like to be cut, sawn or milled from blocks and the need for stocking the moulds for bodies of various shapes is eliminated. Nevertheless, the compressive strength of bodies of foamed glass or the like is increased rather than reduced in comparison with a usual foamed glass body having the same apparent specific gravity because the entire thickness of the material of the cell walls between the macrocells remains the same whereas the microcells impart a lattice-like cross-sectional structure to the cell walls. Besides, the sound insulating properties are improved because the dynamic modulus of elasticity is reduced by the fact that the solid cross-section of the cell walls is divided into smaller solid cross-sections by the microcells. The heat insulation value of the material is also improved by the microcells extending through the cell walls. Another advantage afforded by having microcells in through the cell walls is the fact that the foamed glass or the like is less susceptible to temperature fluctuations so that less care is required in cooling the glass from the fritting temperature.

According to the main patent application porous filler particles may be embedded in the solidified vitreous mass having the foamed structure, whereby the apparent specific gravity may be further reduced and the

[P ]

insulating properties and strength may be further improved, if desired.

5 The process according to the main patent application for producing such a material resides essentially in that ground glass or ground glasslike material is moistened and, if desired, is mixed with the filler in a fine division, and is heated and subsequently cooled in order to impart a foamed structure to the  
10 glass or glasslike material.

It is an object of the present invention to improve the process according to the main patent application of producing a foamed glass, in which microcells extend through the cell walls which separate the macrocells constituting the foamed structure proper. The process according to the present invention resides essentially in that the ground glass is wetted with an aqueous solution which contains alkali metal silicate and an organic substance, the mixture is dried and/or subjected to preliminary roasting, which is effected at a temperature below 600° C., and the mixture is then inflated and cooled in a manner known  
25 per se. The present invention is based on the recognition that the simultaneous use of alkali metal silicate and an organic compound in an aqueous solution renders the success of the process, more particularly the inflating operation, independent of the quality and composition of the ground glass and largely independent of the particle size of the same. Particle sizes of 0.1 to 0.2 mm. may be used without difficulty. As a result, the process  
30 can easily be controlled. When the mixture is being dried, the substances contained in the solution dry on the glass particles so that the inflating substance is present in a fine division in the ground glass during the subsequent heating, which causes the inflation.

Essential advantages will be obtained if the mixture is subjected to preliminary roasting at temperatures below 600° C., at which the inflating effect is not yet obtained. By this treatment, the individual particles of the ground glass are covered with a vitreous crust, in which carbon-containing compounds are embedded in a very fine division. This granulate or powder is then subjected to the actual inflating treatment. Owing to the intimate bond between the various gas-evolving substances and between these substances and the ground glass, the inflation is effected very rapidly, within a few minutes, in contrast to known processes. A longer time is advantageous only to ensure a uniform soaking of the bed of material in dependence on its heat capacity. There are no differences from known processes regarding the subsequent slow cooling of the resulting glass foam. For the sake of higher strength it is recommended to cool as slowly as possible, in dependence on the wall thickness of the resulting bodies, not only during the annealing range of the mixture throughout the cooling step. Grain  
65

sizes of glass powder up to 1.0 mm can be used, if only a certain amount of finely milled glass powder, namely at least 10% by weight, of the whole mixture of glass powder, with a grain size of below 0.035 mm is admixed to the batch. 70

The alkali metal silicate which is employed is preferably a solution of commercially pure water-glass. This solution may be dilute, if desired. To improve the wetting of the ground glass by the water-glass, the latter may have added to it 0.01—0.5 percent by weight, preferably 0.1 percent by weight, of an alkali-resisting wetting agent, such as oxydiphenyldisulphonate. The mixture may be rendered less compact to improve its workability during the roasting operation by the addition of solid, inflated alkali metal silicate, known per se. For instance, 4—30 parts by weight of concentrated water-glass solution and, if desired, up to 5 parts by weight of solid, inflated water-glass are added per 100 parts by weight of ground-glass. 85

It is interesting to note that the addition of substances which usually reduce the viscosity, other than alkali metal silicate, is not successful. For instance, sodium aluminate in a ternary compound with silica tends to promote a refractory bond. Sodium borate is soluble in water only to an amount of 1 1/2 percent in the cold and an admixture of the powder does not appear to result in a sufficiently fine division. Alkali metal phosphates tend to increase the refractoriness of silicate glasses (only pure phosphate glasses are low-melting). Lead oxide reduces the melt viscosity too much so that the gases blow off; besides, it can only be added as a solid, is too heavy and too expensive on a weight basis; it is also poisonous. If substances such as sulphur or nitrate are added to the mixture which influence the combustion of the carbon obtained from the organic substances, the inflation will be inhibited and a light-coloured, sintered or molten product rather than foamed glass will be obtained. If only a small amount is added, for instance, 0.2 % by weight related to ground glass, a white to light grey foamed glass will be obtained. 100

The alkali metal silicate forming the base material of the foam-forming vitreous crust has two functions. It causes the formation of an intimate bond between the covering and the ground glass particle and embeds the very finely divided organic substance so as to exclude air so that this organic substance is cracked and does not begin to evolve carbon monoxide until a temperature above 500—600° C. has been reached. It is believed that this very fine division of the organic substance results in the formation of the microcells in the cell walls of the macrocells during the inflation proper. 110 120 125

It is particularly advantageous to mix the alkali metal silicate with an organic sub- 130

stance which in a very fine division, preferably in a true or colloidal solution or as a highly peptized powder, is compatible with the highly alkaline water-glass and is not sublimated at the roasting temperature. The use of polyhydric alcohols or carbohydrates such as sugar, glycol, glycerol, cellulose, starch or wood flour has proved particularly recommendable. Other water- and/or alkali-soluble substances, such as precondensates of urea formaldehyde or of phenol formaldehyde, or bitumen emulsions, may be used for this purpose and are available at very low cost because they are industrially produced on a very large scale. It is suitable to add 0.3 to 10 parts by weight of organic substances per 100 parts by weight of ground glass.

It is interesting to note that alkali metal silicate and an organic substance alone form a foamable granulate, which seems to be satisfactory but collapses during a subsequent inflating treatment. Any ground glass, however, which is present, will be included in the melt, even if it has a relatively large particle size. The activity is also shown by the fact that perlite spherules, which normally react with glass only at fairly high temperatures, are completely included in the melt in the present case at 800° C. For decorative reasons, however, it is desired that the perlite should be stable at the inflating temperature, which is lower in the present case.

It is characteristic of the process that a glass particle is made first and is covered by a mixing operation with an intimate mixture of such organic substance in an aqueous solution of an alkali metal silicate, and that the subsequent drying or preliminary roasting transforms the aqueous covering into a solid, vitreous crust so that a particle is obtained which contains carbonaceous or carbonizable substances in a very fine division in an outer layer. During a roasting treatment at elevated temperature the carbonizable or carbonaceous substances will be cracked and remain enclosed in the gas-evolving alkali metal silicate crust. Any gases evolved will not escape but remain in the form of minute gas bubbles in the vitreous outer layer on the glass particles. During this roasting treatment, only a very slight sintering is desired and the particles should remain substantially freely movable. It is suitable but not always necessary to subject the resulting particles to a grinding operation, particularly to separate particles which have sintered together.

According to the invention the process may be carried out in such a manner that the moist mixture is heated at a temperature between 300° and 600° C., preferably about 500° C., for 1 to 10 minutes, for instance, in a rotary kiln, and is then cooled to form a pregranulate of agglomerated ground glass particles covered by a crust which contains water-glass and carbon. This pregranulate may be ground

to a particle size below 0.5 mm. before its further processing.

If the pregranulate obtained by the preliminary roasting is crushed to a particle size of, e.g., 1—2 mm., it will have an apparent specific gravity of 0.75 — 0.80 kg./litre. Ground glass which would not usually foam will be foamable up to 300 percent by volume after this treatment. The intimate bond between the foam-forming covering and the ground glass particle, the surface of which already contains diffused alkali metal silicate, reduces the viscosity of the supporting glass so that the actual foaming may be carried out at temperatures which are lower than usual by about 100° C. According to the invention the process may be carried out in such a manner that the pregranulate is heated very rapidly to a temperature between 660° and 760° C. to inflate the mass, which is subsequently slowly cooled. In the process according to the invention the formation and cooling of the pregranulate may be effected in a separate operation, which may be followed by reheating to effect the actual inflation. Admixtures may be added, if desired, to the pregranulate. Alternatively, the two steps of covering the ground glass particles with a foam-forming crust and the formation of a foam-forming granulate and the subsequent actual inflating step may be carried out in an uninterrupted sequence and in the same furnace, if desired. It is essential for the preceding mixing step to result in a uniform wetting of the ground glass particle with the solution of the substances which subsequently form the foam-forming crust.

If it is intended to admix untreated ground glass or other admixtures to the pretreated ground glass before the inflating step, it will be suitable to grind the roasted pregranulate to a particle size below 0.5 mm. before its further processing. In this case up to 30 percent by weight of untreated ground glass, ground foamed glass waste or ground basalt and/or ground slag, and up to 60 percent by volume of light-weight, heat-resisting admixtures, such as expanded mica or perlite, inflated clay or the like, may be admixed. For instance, the activated ground glass allows ground slag having a melting point at 1400° C. to be included in the melt at 780° C. It has proved desirable to wet such additional substances previously with a solution of water-glass in order to ensure a better mixing and adhesion of the foam-forming granulate and the other ground substances to the surface of these admixtures and to avoid a segregation during the movement of the mass through the inflating furnace.

It has been found that an addition of more than two parts by weight of organic substance and more than 15 parts by weight of alkali metal silicate per 100 parts of ground glass results in the formation of a dark grey to black glass foam, the ground waste of which

may be used for repeated inflation instead of the pregranulate. If only ground waste is used, the inflating temperature must be increased by 10—20° C. above that of the first inflating treatment to ensure an expansion. This effect is entirely unknown in connection with the previous foamed glass materials and appears to be due to the fact that the use of a short period at high temperature during the actual inflating step is enabled by the use of the ground glass pretreated according to the invention (pregranulate) and causes only a part of the active portions to be inflated so that the remaining, unused active portions remain available for subsequent inflating processes. This advantage enables a continuous operation, which may be carried out in open moulds, if desired, or without any moulds, because any waste obtained by trimming may be re-used.

The addition of less than one part by weight of organic substance and less than 10 parts by weight of alkali metal silicate per 100 parts of ground glass, however, will result in a white to light grey foamed glass, the ground waste of which has no longer any inflating power and may be admixed to the material to replace the ground glass rather than the foam-forming granulate. Such white to light grey foamed glass may be coloured according to the invention by an addition of heat-resisting inorganic pigments, such as cobalt oxide (cobaltic oxide), chromium oxide (chromic oxide) or titanium oxide (titanium dioxide).

The wetting of the ground glass with the solution containing the alkali metal silicate and the organic substance may be effected, if desired, by mixing in a positive or pan-type mixer in order to ensure an optimum and uniform wetting and distribution.

Foamed glass in the form of blocks or bodies of any desired shape may be obtained by the inflating step. It is not necessary to use closed moulds and pressure. Foamed glass may be obtained in any desired moulds and even in the form of continuous blocks or without any moulds. During the inflating step, the pregranulate agglomerates to form the bodies or blocks. In this way a foamed glass body having the desired shape and size is obtained. If the pregranulate, which may have agglomerated to form particles of different size, is crushed before being inflated, the resulting foamed glass body will have a greater homogeneity.

According to a development of the invention, this agglomeration of the pregranulate can be specifically controlled and promoted. For this purpose the mixture consisting of the ground glass and the aqueous solution containing the alkali metal silicate and the organic substance is pressed while it is still in a moist state to form uniform compacts, which are subjected to preliminary roasting to form a pregranulate.

By the action of heat, the pregranulate is then inflated to form spheroidal foamed glass particles, which may then be subjected to further processing. It has been found that the moist mixture bonded by the alkali metal silicate or water-glass can be shaped in a simple manner to form small compacts, which determine the particle size of the granulate. These compacts may be made, e.g., by extruding the mixture by a screw through a die having a plurality of orifices and dividing the emerging extrusions. These compacts resist deformation in a moist state and are slightly sintered during the preliminary roasting so that these compacts retain their shape and in this state may be subjected to inflation, which will then result in spheroidal foamed glass particles. The moist mixture may be pressed to form compacts 1—2 mm. in diameter. This diameter is increased about five times by inflating and the spherical shape is obtained as the result of the gas pressure in these particles.

Granulating to obtain particles having a predetermined size may also be effected by wetting porous inorganic particles, which are stable at the inflating temperature, such as perlite (inflated lava mineral) or vermiculite (inflated mica), with an aqueous solution which contains alkali metal silicate and an organic substance, followed by dusting with ground glass. Instead of having open cells, as in other cases, the products obtained by roasting and inflating are moisture-tight and even gas-tight and may be used for many purposes for which they were previously unsuitable, for instance, in a mixture with bitumen for the insulation of roofs.

It has proved advantageous to mix ground glass having a coarser particle size, for instance, in a range of 0.1 mm. to 1 mm., with at least 10 percent by weight of finer ground glass having, e.g., a particle size below 0.035 mm. and to mix this mixture with the aqueous solution of the alkali metal silicate and an organic substance. The different particle sizes of the ground glass will then result in a better coherence of the compacts.

Before the preliminary roasting, the compacts may be covered with dry ground glass, for instance, by tumbling. This will reduce the relative moisture content of the compacts before the preliminary roasting to reduce the tendency of the compacts to agglomerate, particularly when a higher moisture content was required for pressing.

To avoid waste, any dust obtained in addition to the foamed glass particles formed from the compacts is wetted with 5—20 percent by weight of a water-glass solution diluted 1:1, and the mixture is re-formed into compacts and subjected to preliminary roasting.

During the inflating treatment proper, the inflated particles should be consolidated in themselves by sintering whereas an agglomeration of the individual particles or compacts

should be avoided. In a preferred embodiment of the invention the compacts are heated to the inflating temperature by contacting them with the surface of a bath, which has been heated to the inflating temperature of 800—900° C., particularly a metal bath, and by moving them relative to the bath surface, if desired. The bath may consist of a bath of lead or aluminium, the latter being preferable. By the application of the compacts to be inflated to a liquid bath surface, the mechanical stress of the particle surface is minimized and agglomeration is avoided. To reliably avoid an agglomeration of the compacts, it is in accordance with the invention to dust the compacts with a powder material which is incompatible with the fusing glass and the molten material of the bath at the inflating temperature, for example, graphite, petroleum coke, Portland cement, sintered corundum, and bentonite, or a mixture of such substances, or the compacts may be embedded into an equal volume of such a powder material.

The compacts may be displaced in the longitudinal direction of the metal bath by means of a conveyor belt which is disposed above the bath surface and provided with scrapers. Alternatively, the inflating compacts disposed on the surface of the bath may be conveyed by a screen, which is disposed below the bath surface and periodically emerges slightly above the surface of the bath. As another alternative, a circulation of the surface of the metal bath may be maintained by electric induction so that the metal bath with the granulate layer is moved through a suitable zone of a furnace.

In the process according to the invention the compacts are suitably maintained adjacent to the bath surface for 0.5 to 5 minutes to heat the compacts. If the compacts are displaced in steps over the bath surface during this time, it is suitable to give the bath an elongated configuration. The compacts are desirably moved over the bath surface in a layer having a height of one particle. However, if an agglomeration of the compacts has been prevented by dusting them with suitable materials, the layer of compacts may be higher. In this case it is desirable, however, to provide for a motion within the layer. The bath being contained in a closed furnace, the temperature required for inflating prevails also above the bath surface. Because the individual compacts are inflated to five times their diameter, the height of the layer will be automatically increased if the individual compacts are applied to the bath surface in a closely spaced arrangement at the beginning of the process. Owing to the increase of the volume of the inflating particles, the same will tend to pile up. If this is to be avoided, the uninflated particles must not cover the entire bath surface at the beginning of the process. To effect a slow cooling in the annealing range of the

glass, it has been found suitable to move the foamed glass particles for a short time, for instance, 1—10 minutes, through a zone which is so heated that the particles are cooled somewhat (20—30° C.) below the strain point of the glass (e.g., 520—530° C.). The foamed glass particles thus obtained are largely free of internal stresses and for this reason are particularly suitable for the manufacture of insulating materials having a foamed structure and containing such foamed glass particles embedded in foamed plastics. If the foamed glass particles are inflated to form bodies, such as slabs, the foamed glass particles may be quenched because the further inflating will in any case cause the foamed glass particles to agglomerate. It is preferable, however, not to quench the foamed glass particles but to introduce them in a hot condition into the moulds and to move the charged moulds through an inflating furnace, in which the foamed glass particles are further inflated to agglomerate and fill the mould.

In the spheroidal foamed ground glass particles made according to the invention the cell walls are filled with microcells and the outer skin of the spherules is not thicker than an internal cell wall and may even contain microcells. The same applies to the covering of particles made by granulating with the aid of inorganic porous bodies, such as perlite (inflating lava mineral) or vermiculite (inflated mica), with the difference that such spheroidal particles contain a core of the foreign matter. The spheroidal foamed glass particles made according to the invention can be used for manifold purposes. They may be used in bulk as a filler for cavities to provide a good insulation against heat and sound. They may also be used for making insulating material in the form of slabs, blocks etc. For instance, such spheroidal foamed glass particles may be embedded in a glass foam in order to form an insulating body, in which the glass foam contains spherical foamed glass particles having a low apparent specific gravity and a high insulation value. For this purpose the foamed glass particles, either as such or with a covering of ground glass and an aqueous solution containing alkali metal silicate and an organic substance, are charged in bulk into open or closed moulds and re-heated so that they are inflated and agglomerate to form a moulded body. The foamed glass particles are suitably covered before heating and charging into the mould by superficially wetting them with an aqueous solution containing alkali metal silicate and an organic substance, followed by dusting with ground glass. As is known from the literature, the material is considered to have a certain memory. Because the agglomerated particles are different in spite of the fact that the basic material is the same, the resulting moulded body may be considered to have a certain degree of heterogeneity. This

- results in an improved resistance to cyclic changes of temperature compared to homogeneous foamed glass bodies so that the inflated bodies may be cooled rapidly without setting up excessive internal stresses, which would result in cracks. This is particularly applicable to foamed glass particles which have been granulated around inorganic, porous particles, such as perlite (inflated lava mineral) or vermiculite (inflated mica).
- The spheroidal foamed glass particles according to the invention may be embedded to special advantage in a material of different kind, such as synthetic resin, particularly a synthetic resin foam. For this reason an insulating material comprising spheroidal foamed glass particles made by the present process is essentially characterized according to the invention in that the foamed glass particles are embedded in a synthetic resin foam, particularly in a foam of polystyrene, a phenolic resin, polyurethane, polyester or polyether. The proportions of the synthetic resin foam and the spherical foamed glass particles may vary so that the synthetic resin or the spheroidal foamed glass particles may constitute a major part of the insulating material. A particularly good insulating material will be obtained according to the invention if the synthetic resin foam is contained in the insulating material only in that amount which is required for bonding the foamed glass particles so that this insulating material consists substantially of the spheroidal foamed glass particles and the interstices between these particles are filled by the synthetic resin foam.
- Such an insulating material which contains foamed glass particles embedded in a synthetic resin foam is distinguished by a particularly high insulation value. Compared to an insulating material which consists only of synthetic resin foam it has the advantage of higher strength, improved heat resistance and reduced shrinkage. Compared to an insulating material consisting only of foamed glass it has the advantage of higher elasticity owing to the resilient embedding of the foamed glass particles in synthetic resin foam and lower apparent specific gravity because the synthetic resin foam is usually lighter in weight than the glass foam.
- The spheroidal foamed glass particles embedded in the synthetic resin foam have generally a diameter of about 1—10 mm. Glass spherules or particles of the usual foamed glass would render a machining of the insulating material by sawing or drilling or boring very difficult or would even prevent such machining, particularly if such particles or spherules are closely spaced. The foamed glass particles made according to the invention, however, have microcells in the cell walls between the macrocells and the structure of such particles presents virtually no resistance to machining because the micropores in the cell walls define points of intentional weakness and readily enable a machining of the foamed glass.
- EXAMPLE 1**
- 10 kg. ground glass made by grinding bottle glass to 0—0.2 mm. and 0.25 kg. inflated alkali metal silicate having an apparent specific gravity of about 0.06 kg./litre are intimately mixed with a solution consisting of 2 kg. water-glass having a density of 1.4, 1.5 litre water, 0.3 kg. sugar and 30 grams wetting agent (Benax 2 A 1). (Registered Trade Mark). The mixture is heated in a rotary kiln at about 550° C. for 5 minutes, rapidly cooled and ground to less than 0.5 mm. A dark grey powder is obtained, in which all glass particles are covered with a foamlike crust.
- This grey to black foam-forming granulate is charged in an open mould of iron into a furnace heated to about 700° C. and is heated to 730° C. and held at this temperature for one hour. After a slow cooling for about 18 hours, the material is removed from the furnace at about 50° C. and removed from the mould.
- A dark grey to black foamed glass body is obtained, which does not develop an odour when being sawn apart and under the microscope exhibits a highly uniform structure with numerous microcells in the internal cell walls.
- EXAMPLE 2**
- 2 kg. water-glass, 1.5 litre water, 0.3 kg. sugar and 30 grams wetting agent are mixed with 0.25 kg. inflated alkali metal silicate and granulated in a rotary kiln. This granulate is ground, mixed with 10 kg. ground glass and heat-treated as in Example 1. This results only in a loose powder.
- EXAMPLE 3**
- The foamed glass bodies made according to Example 1 are ground to a particle size of 0.75 mm. and heated as described in Example 1 to 750° C. The characteristics of the resulting body are similar to those described in Example 1.
- EXAMPLE 4**
- 6 parts by weight of the foam-forming granulate made according to Example 1 are intimately mixed with 4 parts by weight of untreated ground glass and heated as described in Example 1 to 740° C. A good foamed glass is also obtained.
- EXAMPLE 5**
- 10 kg. ground glass are intimately mixed with a solution consisting of 1 kg. water-glass, 2 litres water, 0.1 kg. sugar and 20 grams wetting agent and with 0.4 kg. inflated alkali metal silicate and processed as described in Example 1 to form a foam-forming granulate. This is somewhat lighter in colour. The inflating of this foam-forming granulate as de-



scribed in Example 1 results in a light-grey foamed glass. Grinding this product and reheating it to 750° C. will not result in inflation.

#### EXAMPLE 6

10 kg. ground glass are thoroughly wetted with a solution of 2.5 kg. water-glass, 1 litre water, 0.2 kg. sugar and 20 grams wetting agent and processed to form foamed glass directly by heating to 730° C.

#### EXAMPLE 7

10 kg. ground glass are mixed with a mixture of 0.9 kg. water-glass and 0.3 kg. concentrated glycerol and processed as described in Example 6 to form foamed glass. The resulting bodies are particularly dark and have a large number of microcells in the internal cell walls.

#### EXAMPLE 8

10 kg. ground glass and 0.25 kg. inflated alkali metal silicate are processed with a mixture consisting of 1.5 kg. water-glass, 1.5 litre water, 0.5 kg. fine wood flour and 30 grams wetting agent and are inflated by a heat treatment as described in Example 6. The resulting foamed glass is somewhat lighter in colour and has coarser cells than that obtained in Example 1.

#### EXAMPLE 9

10 kg. ground glass and 0.25 kg. inflated alkali metal silicate are mixed with a solution of 1.5 kg. water-glass, 1.5 litre water, 0.3 kg. alcohol-soluble phenolic resin and 30 grams wetting agent and roasted to form a pregranulate. Inflating is effected as described in Example 1.

#### EXAMPLE 10

10 kg. ground glass and 0.25 kg. inflated alkali metal silicate are processed with a mixture consisting of 1.5 kg. water-glass, 1.5 litre water, 30 grams wetting agent and 0.5 kg. of an alkali-resisting bitumen emulsion and roasted to form a pregranulate. Inflating results in a foamed glass, which is somewhat lighter in colour.

#### EXAMPLE 11

7 kg. of the pregranulate made according to Example 1 are mixed with 3 kg. vermiculite which has a particle size of 2—5 mm. and has been moistened with a mixture of 0.7 kg. water-glass and 1 litre water. The vermiculite particles are thus covered with the foam-forming granulate. After drying, inflating is effected at 740° C. in open moulds of iron for one hour. A body similar to rock is obtained. When this body, which contains vermiculite, has been ground, the material cannot be re-inflated alone.

#### EXAMPLE 12

6 kg. of the foam-forming granulate made according to Example 1 are mixed with 4 kg. perlite, which has been pretreated with 0.3 kg. waterglass and 0.5 litre water. The mixture is dried. Inflating as described in Example 1 results in a body similar to rock.

#### EXAMPLE 13

4 kg. of a foam-forming granulate produced according to Example 1 are mixed with 6 kg. of the ground, perlite-containing foamed glass made according to Example 12 and the mixture is heated as described in Example 1 to 700° C. The resulting surface has a very high permeability to gas (about 1000 nanoperm) and an unsatisfactory strength and may be used for filtering purposes.

#### EXAMPLE 14

5 kg. foam-forming granulate according to Example 1 are intimately mixed with 5 kg. inflated clay, which has been moistened with 0.3 kg. water-glass and 0.2 litres water. The mixture is heated according to Example 1 to 720° C. The resulting body is somewhat heavier and has a very high mechanical strength and a lower permeability to gas.

#### EXAMPLE 15

10 kg. ground glass and 0.3 kg. inflated alkali metal silicate are processed according to Example 1 with a solution consisting of 1 kg. water-glass, 2 litres water, 0.1 kg. sugar and 30 grams wetting agent to form a pregranulate.

1 kg. of this pregranulate is inflated according to Example 1 with 0.5 kg. ground glass and 1 kg. perlite, with an addition of 30 grams cobaltic oxide. The resulting body has an optical activity and comprises white particles embedded in a blue, vitreous matrix. This body is highly suitable for decorative purposes.

#### EXAMPLE 16

10 kg. ground glass are thoroughly wetted with a solution of 2.5 kg. water-glass, 1 litre water, 0.2 kg. sugar and 20 grams wetting agent. The resulting pulpy mixture is granulated, for instance, by extruding the mixture by a screw through a multiple orifice die and cutting the emerging extrusions apart. This results in particles about 1—2 mm. in diameter. These particles are heated in a rotary kiln at a temperature between 300° and 600° C., approximately 400° C., for 2 minutes and are then cooled. This results in a slight sintering, by which a certain resistance to deformation is imparted to the particles. The granulate which has thus been subjected to preliminary roasting is embedded in the same volume of Portland cement in order to avoid an agglomeration during the subsequent inflating step. This mixture is

then applied to the surface of a liquid aluminium bath having a temperature of 780—790° C. and in a layer having a height of one particle is displaced in steps on the bath surface. The granulate is maintained in contact with the bath surface for about 3 minutes. The action of heat causes the individual particles of the granulate to inflate to spheroidal foamed glass particles, which have an inflated diameter of 5—10 mm. These foamed glass spherules are then cooled. Cooling may be effected in contact with the air and it is not necessary to adopt special steps, such as the use of tunnel kilns, to ensure a slow cooling. This thermal resistance of the foamed glass particles is due to the fact that the cell walls between the macrocells have microcells so that the cell walls can take up the thermal stresses set up during a rapid cooling. The spherical foamed glass particles thus obtained may be used for various purposes.

#### EXAMPLE 17

1 kg. foamed polystyrene resin product, such as "Styropor" (Registered Trade Mark) is intimately mixed with 10 kg. of the spherical foamed glass particles made according to Example 16 and 500 grams of a known adhesive. This mixture is charged into a mould, for instance, for the manufacture of slabs. The Styropor is then inflated in known manner by the action of heat and is thereby caused to bond to the foamed glass particles.

DATA OF FOAMED GLASS SPECIMENS MADE ACCORDING TO THE PRECEDING EXAMPLES

Example	Apparent Specific Gravity	Tensile Strength in Bending	Com- pressive Strength	Thermal Con- ductivity	Permea- bility to Gas	Water Absorption Capacity
	kg./litre	kg./sq. cm.	kg./sq. cm.	kcal./m. h. °C.	nanoperm	percent by volume
1	0.20	8	18	0.06	7	1.6
3	0.22	9	25	—	—	—
4	0.22	9	20	0.065	22	2.1
5	0.23	11	25	0.07	16	1.7
6	—	13	26	0.07	17	2.0
7	0.18	10	18	0.55	12	1.9
8	0.24	11	24	0.07	20	2.5
9	0.25	13	28	0.075	20	2.3
10	0.26	14	27	0.075	19	2.8
11	0.25	8	16	0.075	220	12.5
12	0.24	9	21	0.07	95	10.2
14	0.40	27	65	0.11	21	6.1

#### WHAT I CLAIM IS:—

1. A process of producing an insulating material as claimed in claim 1 of Application No. 16582/62 (Serial 1,000,136), in which ground glass is wetted with an aqueous solution or suspension which contains alkali metal silicate and an organic substance, the mixture is dried and/or subjected to preliminary roasting, which is effected at a temperature below 600°C., and the mixture is then inflated and cooled in a manner known per se.
2. A process according to claim 1, characterized in that 4—30 parts by weight of concentrated water-glass solution are added per

100 parts by weight of ground glass.

3. A process according to claim 1 or 2, characterized in that 0.01—0.5 percent by weight of an alkali-resisting wetting agent are added to the water-glass.

4. A process according to any of claims 1 to 3, characterized in that polyhydric alcohols or carbohydrates which are compatible with alkali metal silicate in solution and are not sublimated at the roasting temperature, such as sugar, glycol, glycerol, cellulose, starch or other water and/or alkali-soluble substances, such as precondensates of urca formaldehyde or of phenol formaldehyde, or bitumen emul-

sions, are used as the organic substance.

5. A process according to claim 4, characterized in that 0.3 to 10 parts by weight of organic substances are added per 100 parts by weight of ground glass.

6. A process according to any of claims 1 to 5, characterized in that ground glass having a particle size up to 1.0 mm is mixed with at least 10 percent by weight of ground glass having a smaller particle size, e.g., less than 0.035 mm., and the aqueous solution of an alkali metal silicate and an organic substance.

7. A process according to any of claims 1 to 6, characterized in that the pregranulate which has been subjected to preliminary roasting is ground to a particle size below 0.5 mm. before its further processing.

8. A process according to any of claims 1 to 7, characterized in that the pregranulate is very rapidly heated to a temperature between 660°C and 760°C to inflate the mass, and is subsequently slowly cooled.

9. A process according to any of claims 1 to 8, characterized in that the pregranulate in a mixture with untreated ground glass, ground foamed glass waste, ground basalt and/or ground slag is very rapidly heated to a temperature of 660° — 760°C to inflate the mass, and is subsequently slowly cooled.

10. A process according to any of claims 1 to 9, characterized in that more than 2 parts by weight of the organic substance and more than 15 parts by weight of alkali metal silicate are added with the result then that a dark grey to black foamed glass is obtained, the ground waste of which may be subjected to re-inflation instead of the pregranulate.

11. A process according to any of claims 1 to 10, characterized in that less than 1 part by weight of organic substance and less than 10 parts by weight of alkali metal silicate are added with the result that a white to light grey foamed glass is obtained.

12. A process according to any of claims 1 to 6, 8, 10 and 11, characterized in that the moist mixture is pressed to form uniform, small compacts, which are subjected to preliminary roasting to form a pregranulate, and the granulate formed by the compacts is then inflated by the action of heat to form spheroidal foamed glass particles, which may be subjected to further processing.

13. A process according to any of claims 1 to 12, characterized in that the moist mixture is or the compacts are heated at a temperature between 300° and 600° C., preferably about 500° C. for 1—10 minutes, for instance, in a rotary kiln, and then cooled to form a pregranulate of agglomerated ground glass particles covered by a crust which contains water-glass and carbon.

14. A process according to claim 12 or 13, characterized in that the compacts are covered with a fine, dry, ground glass, for instance,

by tumbling, before the preliminary roasting.

15. A process according to any of claims 1 to 14, characterized in that granulating is effected in that porous inorganic particles which are stable at the inflating temperature, such as perlite (inflated lava mineral or vermiculite (inflated mica), are covered with the mixture of ground glass and an aqueous solution containing alkali metal silicate and an organic substance and are then inflated.

16. A process according to any of claims 12 to 15, characterized in that the compacts are contacted with the surface of a metal bath which has been heated to the inflating temperature of 800—900° C., particularly a metal bath, and are preferably moved relative to the bath surface.

17. A process according to claim 16, characterized in that the compacts are maintained adjacent to the bath surface for 0.5 to 5 minutes.

18. A process according to claim 16 or 17, characterized in that the compacts are displaced over the quiescent bath surface.

19. A process according to claim 16 or 17, characterized in that a circulation of the bath surface is maintained by electric induction.

20. A process according to any of claims 16 to 19, characterized in that a material which is not compatible with the fusing glass and the molten material of the bath at the inflating temperature, for example, graphite, petroleum coke, Portland cement, sintered corundum, or bentonite, or a mixture thereof, is applied to the compacts.

21. A process according to any of claims 16 to 20, characterized in that the compacts are moved over the bath surface in a layer which has a height of one particle.

22. A process according to any of claims 12 to 21, characterized in that the foamed glass particles formed from the compacts are moved for a short time, for instance, 1—10 minutes, preferably about 5 minutes, through a zone which is so heated that the particles are cooled somewhat (20—30° C.) below the strain point of the glass (e.g., 520—530° C.) in order to effect a slow cooling in the annealing range.

23. A process according to any of claims 12—22, characterized in that the foamed glass particles formed from the compacts are loosely charged into an open or closed mould and are reheated so as to be re-inflated and to agglomerate and form a body of foamed glass.

24. A process according to any of claims 12—22, characterized in that the foamed glass particles formed from the compacts are wetted with an aqueous solution containing alkali metal silicate and an organic substance and are dusted with ground glass and then loosely charged into an open or closed mould and reheated, whereby they are caused to be re-inflated and to agglomerate and form a body of foamed glass.

25. A process according to any of claims 12 to 24, characterized in that dust which is obtained in addition to the foamed glass particles formed from the compacts is wetted with  
5 5—20 percent by weight of a water-glass solution diluted 1:1 and the mixture is reformed into compacts and subjected to preliminary roasting.
26. Spheroidal foamed glass particles produced by a process according to any of claims 12 to 25, characterized in that they have a uniform pore structure substantially throughout their cross-section and their outer skin is not thicker than an internal pore wall.
- 10 27. Foamed glass particles produced by the process of claim 15, characterized in that inorganic porous particles, such as perlite (inflated lava mineral) or vermiculite (inflated

mica) are covered by a gas-tight foamed glass crust which contains macrocells and microcells in the macrocell walls.

28. An insulating material manufactured from spheroidal foamed glass particles according to claim 26 or 27, characterized in that the foamed glass particles are embedded in a synthetic resin foam, particularly in a foam of polystyrene, a phenolic resin, polyurethane, polyester or polyether foam.

29. An insulating material according to claim 28, characterized in that it contains the synthetic resin foam only in the amount required for bonding the foamed glass particles.

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